PATENT
CASE 4233C3

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants:	Donald B. Appleby et al	:	Group Art Unit: 1211
Serial No.:	08/360,184	:	Examiner: E. White
Filed:	December 20, 1994	:	
For:	**Polyol Polyester Synthesis**		

DECLARATION UNDER 37 C.F.R. §1.608 OF ROGER S. BERGER

Assistant Commissioner for Patents
Washington, DC 20231

Dear Sir:

I, ROGER S. BERGER, declare that:

1. I have been employed by the assignee of the present application, The Procter & Gamble Company, since January 1990. From January 1989 until my employment with The Procter & Gamble Company in January 1990, I worked at The Procter & Gamble Company through a contract agency, Marketing Programs.

2. I have a Bachelor in Chemistry Degree from Miami University, Oxford, Ohio.

3. From January 16, 1989 to the present, I have worked as a Senior Research Associate on the sucrose polyester synthesis project. I have conducted sucrose polyester laboratory experiments wherein sucrose and fatty acid methyl ester were reacted to form sucrose fatty acid polyesters (sucrose polyesters) according to predetermined test plans and analyzed the results (or had the results analyzed under my direction and control) according to established procedures.

4. I conducted and analyzed a variety of sucrose polyester laboratory experiments between February 1989 and May 1990 under the direction and control of Mr. Patrick Corrigan, including those described in this Declaration.

5. For each experiment which I conducted and which is described in this Declaration, I accurately recorded, on or about the day of the experiment, the general nature of the experiment, including pertinent reaction parameters, the results of the experiment, the date of the experiment and my signature in a Laboratory Notebook assigned to me for such a purpose.

6. I have examined Exhibit 28, and I confirm that Exhibit 28 comprises accurate copies of pages 34-39 of Laboratory Notebook SI 1384 on which I accurately recorded a series of experiments I conducted in September 1989 using a laboratory scale continuous reactor system, and which I signed and accurately dated upon the completion of the procedures described on each page respectively. Laboratory Notebook SI 1384 was in my possession and control from June 1989 to October 1991.

7. Pages 34-37 of Exhibit 28 describe a sucrose polyester reaction which I conducted on September 5, 7 and 8, 1989 and which was part of several experiments intended to evaluate, inter alia, the effect of various sucrose particle sizes on the sucrose polyester reaction. Page 34 sets forth that the sucrose reactant was milled and sieved so that the particle size of the sucrose used in the reaction was less than about 63 microns. Lines 4-15 of page 36 detail the percentages of the respective sucrose esters (i.e., monoester through octaester, represented as 1-8, respectively) in and the average degree of esterification based on mole fractions, I-bar, of the sucrose polyester product of this reaction and the percentage of unreacted sucrose in the reaction mixture as a function of time beginning at 1.5 hours into the reaction. As indicated on page 36, the sucrose polyester product comprised an average degree of esterification of about 2.77 and the reaction mixture comprised about 5.11% unreacted sucrose, all at 4.5 hours into the reaction.

8. Pages 38 and 39 of Exhibit 28 describe a sucrose polyester reaction which I conducted on September 19 and 20, 1989 and which also was part of the experiments intended to evaluate, inter alia, the effect of various sucrose particle sizes on the sucrose polyester reaction. Page 38 sets forth that the sucrose reactant was milled and sieved so that the particle size of the sucrose used in the reaction was less than about 38 microns. Lines 25-29 of page 39 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction and the percentage of unreacted sucrose in the reaction mixture as a function of time beginning at 1.5 hours into the reaction. As indicated on page 39, the sucrose polyester product

had an average degree of esterification of about 2.97 and the reaction mixture comprised about 3.78% unreacted sucrose at 4.5 hours into the reaction.

9. I have examined Exhibit 29, and I confirm that Exhibit 29 comprises accurate copies of pages 89-93 and 96 of Laboratory Notebook SI 6044 on which I accurately recorded a series of experiments I conducted in November 1989, and which I signed and accurately dated upon the completion of the procedures described on each page respectively. Laboratory Notebook SI 6044 was in my possession and control from January 1989 to November 1993.

10. Pages 89-91 and 96 of Exhibit 29 describe sucrose polyester reactions which I conducted on November 3, 6, 7, 11 and 15, 1989 and which were intended to evaluate, inter alia, the effect of various catalyst amounts on the difatty ketone (DFK) content and I-bar of the sucrose polyester product. Pages 92-93 of Exhibit 29 describe sucrose polyester reactions which I conducted on November 8 and 10, 1989 and which were intended to evaluate, inter alia, the effect of various soap amounts on the difatty ketone (DFK) content and I-bar of the sucrose polyester product.

11. I have examined Exhibit 30, and I confirm that Exhibit 30 comprises accurate copies of pages 76 and 78-81 of Laboratory Notebook SI 1384 on which I accurately recorded a series of experiments I conducted in December 1989 using a laboratory continuous reactor system, and which I signed and accurately dated upon the completion of the procedures described on each page respectively.

12. Page 78 of Exhibit 30 describes a sucrose polyester reaction which I conducted on December 21, 1989 and which was part of several experiments intended to evaluate, inter alia, the effect of various sucrose particle sizes on the sucrose polyester reaction. Page 78 indicates that the sucrose reactant used in the reaction was the same sucrose described on page 76 of Exhibit 30. Page 76 of Exhibit 30 indicates that the sucrose was powdered by jet milling. Based upon my experience and recollection, the jet milling procedures used to produce the sucrose reactant described on page 76 typically yielded sucrose particle sizes of about 10 microns or less. Lines 24-30 of page 78 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction and the percentage of unreacted sucrose in the reaction mixture as a function of time beginning at 1.5 hours into the reaction. As indicated on page 78, the sucrose polyester product comprised an average degree of esterification of about 3.20 and the reaction mixture comprised about 0.63% unreacted sucrose at 4.5 hours into the reaction.

13. Pages 79 and 80 of Exhibit 30 describe a sucrose polyester reaction which I conducted on December 27 and 28, 1989 and which was part of the experiments intended to evaluate, inter alia, the effect of various sucrose particle sizes on the sucrose polyester reaction. Page 79 sets forth that the sucrose reactant used in the reaction was jet milled. Based upon my experience and recollection, the jet milling procedures used to produce the sucrose reactant described on page 79 typically yielded sucrose particle sizes of about 10 microns or less. Lines 24-30 of page 80 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction and the percentage of unreacted sucrose in the reaction

mixture as a function of time beginning at 1.5 hours into the reaction. As indicated on page 80, the sucrose polyester product comprised an average degree of esterification of about 2.86 and the reaction mixture comprised about 1.13% unreacted sucrose at 4.5 hours into the reaction.

14. Page 81 of Exhibit 30 describes a sucrose polyester reaction which I conducted on December 29, 1989 and which was also part of the experiments intended to evaluate, inter alia, the effect of various sucrose particle sizes on the sucrose polyester reaction. Page 81 sets forth that the sucrose reactant used in the reaction was the same sucrose described on page 79 of Exhibit 30, which was jet milled. As set forth in paragraph 11, based upon my experience and recollection, the jet milling procedures used to produce the sucrose reactant described on page 79 typically yielded sucrose particle sizes of about 10 microns or less. Lines 24-30 of page 81 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction and the percentage of unreacted sucrose in the reaction mixture as a function of time beginning at 1.5 hours into the reaction. As indicated on page 81, the sucrose polyester product comprised an average degree of esterification of about 2.84 and the reaction mixture comprised about 0.71% unreacted sucrose at 4.5 hours into the reaction.

15. I have examined Exhibit 31, and I confirm that Exhibit 31 comprises accurate copies of pages 24, 25, 28, 32, 33 and 45-47 of Laboratory Notebook SI 6055 on which I accurately recorded a series of experiments I conducted in February and March of 1990, and which I signed and accurately dated upon the completion of the procedures described on each

page respectively. Laboratory Notebook SI 6055 was in my possession and control from November 1989 to June 1990.

16. Page 24 of Exhibit 31 describes a sucrose polyester reaction experiment which I conducted on February 7, 1990 and which was part of several experiments intended to evaluate, inter alia, the effect of reaction pressure on a sucrose polyester reaction employing nitrogen sparging. Page 24 sets forth the temperature and pressure of the reaction at thirty (30) minute intervals, beginning at 7:55 a.m. The reaction pressure was held constant at about 15 mm Hg during the reaction. Lines 23-29 of page 24 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours into the reaction. Also set forth on page 24 in the column labeled "DFK" are the difatty ketone contents of the product, also as a function of time, in ppm. As indicated on page 24, the sucrose polyester product comprised about 90.3% octaester, had an average degree of esterification of about 7.86, and comprised about 308 ppm DFK, all at 6.5 hours.

17. Page 25 of Exhibit 31 describes a sucrose polyester reaction experiment which I conducted on February 8, 1990 and which was also part of the experiments intended to evaluate, inter alia, the effect of reaction pressure on a sucrose polyester reaction employing nitrogen sparging. Page 25 sets forth the temperature and pressure of the reaction at thirty (30) minute intervals, beginning at 8:05 a.m. The reaction pressure was held constant at 15 mm Hg until 9:45 a.m., at which point the reaction pressure was increased from 15 mm Hg to 25 mm Hg. Lines 23-29 of page 25 detail the percentages of the respective sucrose esters in and the I-bar of

the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours into the reaction and the DFK content of the product, also as a function of time, in ppm. As indicated on page 25, the sucrose polyester product comprised about 88.4% octaester, had an average degree of esterification of about 7.85, and comprised about 263 ppm DFK, all at 6.5 hours.

18. Page 28 of Exhibit 31 describes a sucrose polyester reaction experiment which I conducted on February 13, 1990 and which was also part of the experiments intended to evaluate, inter alia, the effect of reaction pressure on a sucrose polyester reaction employing nitrogen sparging. Drop wise addition of the fatty acid methyl ester was also employed in a later stage of the reaction. Page 28 sets forth the temperature and pressure of the reaction at thirty (30) minute intervals, beginning at 8:00 a.m. The reaction pressure was held constant at 15 mm Hg. Lines 23-30 of page 28 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours into the reaction and the DFK content of the product, also as a function of time, in ppm. As indicated on page 28, the sucrose polyester product comprised about 74.2% octaester, had an average degree of esterification of about 7.68, and comprised about 75 ppm DFK, all at 7.5 hours.

19. Pages 32 and 33 of Exhibit 31 describe a sucrose polyester reaction which I conducted on February 20 and 21, 1990 and which was also part of the experiments intended to evaluate, inter alia, the effect of reaction pressure on a sucrose polyester reaction employing nitrogen sparging. Pages 32 and 33 set forth the approximate temperature and pressure of the reaction at thirty (30) minute intervals, beginning at 8:00 a.m. The reaction pressure was held

constant at about 15 mm Hg until 9:35 a.m., at which point the reaction pressure was increased from about 15 mm Hg to about 25 mm Hg. Lines 10-23 of page 33 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours into the reaction and the DFK content of the product, also as a function of time, in ppm. As indicated on page 33, the sucrose polyester product comprised about 85.7% octaester, had an average degree of esterification of about 7.81, and comprised about 243 ppm DFK, all at 13.5 hours.

20. Page 45 of Exhibit 31 describes a sucrose polyester reaction which I conducted on March 9, 1990 and which was also part of the experiments intended to evaluate, inter alia, the effect of reaction pressure on a sucrose polyester reaction employing nitrogen sparging. Page 45 sets forth the approximate temperature and pressure of the reaction at thirty (30) minute intervals, beginning at 8:00 a.m. The reaction pressure was held constant at about 15 mm Hg until 9:40 a.m., at which point the reaction pressure was increased from about 15 mm Hg to about 30 mm Hg. Lines 23-30 of page 45 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours into the reaction and the DFK content of the product, also as a function of time, in ppm. As indicated on page 45, the sucrose polyester product comprised about 71.0% octaester, had an average degree of esterification of about 7.61, and comprised about 96 ppm DFK, all at 7.5 hours.

21. Pages 46 and 47 of Exhibit 31 describe a sucrose polyester reaction which I conducted on March 12 and 13, 1990 and which was also part of the experiments intended to evaluate, inter alia, the effect of reaction pressure on a sucrose polyester reaction employing nitrogen sparging. Page 46 sets forth the approximate temperature and pressure of the reaction at thirty (30) minute intervals, beginning at 7:55 a.m. The reaction pressure was held constant at about 15 mm Hg until 9:35 a.m., at which point the reaction pressure was increased from about 15 mm Hg to about 45 mm Hg. Lines 4-15 of page 47 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours into the reaction and the DFK content of the product, also as a function of time, in ppm. As indicated on page 47, the sucrose polyester product comprised about 75.7% octaester, had an average degree of esterification of about 7.66, and comprised about 137 ppm DFK, all at 14.5 hours.

22. I have examined Exhibit 32, and I confirm that Exhibit 32 comprises accurate copies of pages 29, 30, 36-38 and 86-88 of Laboratory Notebook SI 6055 on which I accurately recorded a series of experiments I conducted in February and May of 1990, and which I signed and accurately dated upon the completion of the procedures described on each page respectively.

23. Page 29 of Exhibit 32 describes a sucrose polyester reaction which I conducted on February 13, 1990 and which was part of several experiments intended to evaluate, inter alia, the effect of alternate catalysts on the sucrose polyester reaction. Page 29 sets forth that sodium methoxide was used as the catalyst for this reaction. Lines 23-28 of page 29 detail the

percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction and the percentage of unreacted sucrose in the reaction mixture as a function of time beginning at 1.5 hours into the reaction. The ppm of DFK in the product are also set forth. As indicated on page 29, the sucrose polyester product comprised about 70.3% octaester, had an average degree of esterification of about 7.61, and comprised about 179 ppm DFK, all at 5.5 hours into the reaction.

24. Page 30 of Exhibit 32 describes a sucrose polyester reaction which I conducted on February 15, 1990 and which was part of the experiments intended to evaluate the effect of alternate catalysts on the sucrose polyester reaction. Page 30 sets forth that sodium methoxide was used as the catalyst for this reaction. Lines 23-28 of page 30 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction and the percentage of unreacted sucrose in the reaction mixture as a function of time beginning at 1.5 hours into the reaction. The ppm of DFK in the product are also set forth. As indicated on page 30, the sucrose polyester product comprised about 86.4% octaester, had an average degree of esterification of about 7.82, and comprised about 221 ppm DFK, at 6.5 hours into the reaction.

25. Pages 36 and 37 of Exhibit 32 describe a sucrose polyester reaction which I conducted on February 26 and 27, 1990 and which was also part of the experiments intended to evaluate, inter alia, the effect of alternate catalysts on the sucrose polyester reaction. Page 36 sets forth that sodium methoxide was used as the catalyst for this reaction. Lines 13-27 of page 37 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester

product of this reaction and the percentage of unreacted sucrose in the reaction mixture as a function of time beginning at 1.5 hours into the reaction. As indicated on page 37, the sucrose polyester product comprised about 73.8% octaester and had an average degree of esterification of about 7.67 at 14.5 hours into the reaction.

26. Page 38 of Exhibit 32 describes a sucrose polyester reaction which I conducted on February 28, 1990 and which was also part of the experiments intended to evaluate, inter alia, the effect of alternate catalysts on the sucrose polyester reaction. Page 38 sets forth that sodium methoxide was used as the catalyst for this reaction. Lines 23-28 of page 38 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction and the percentage of unreacted sucrose in the reaction mixture as a function of time beginning at 1.5 hours into the reaction. As indicated on page 38, the sucrose polyester product comprised about 55.9% octaester and had an average degree of esterification of about 7.36 at 5.5 hours into the reaction.

27. Page 86 of Exhibit 32 describes a sucrose polyester reaction which I conducted on May 29, 1990 and which was also part of the experiments intended to evaluate, inter alia, the effect of alternate catalysts on the sucrose polyester reaction. Page 86 sets forth that potassium methoxide was used as the catalyst for this reaction. Lines 23-28 of page 86 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction and the percentage of unreacted sucrose in the reaction mixture as a function of time beginning at 1.5 hours into the reaction. As indicated on page 86, the sucrose polyester

product comprised about 82.0% octaester and had an average degree of esterification of about 7.75 at 5.5 hours into the reaction.

28. Page 87 of Exhibit 32 describes a sucrose polyester reaction which I conducted on May 30, 1990 and which was also part of the experiments intended to evaluate, inter alia, the effect of alternate catalysts on the sucrose polyester reaction. Page 87 sets forth that potassium methoxide was used as the catalyst for this reaction. Lines 23-29 of page 87 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction and the percentage of unreacted sucrose in the reaction mixture as a function of time beginning at 1.5 hours into the reaction. As indicated on page 87, the sucrose polyester product comprised about 84.7% octaester and had an average degree of esterification of about 7.77 at 6.5 hours into the reaction.

29. Page 88 of Exhibit 32 describes a sucrose polyester reaction which I conducted on May 31, 1990 and which was also part of the experiments intended to evaluate, inter alia, the effect of alternate catalysts on the sucrose polyester reaction. Page 88 sets forth that potassium methoxide was used as the catalyst for this reaction. Lines 23-28 of page 88 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction and the percentage of unreacted sucrose in the reaction mixture as a function of time beginning at 1.5 hours into the reaction. As indicated on page 88, the sucrose polyester product comprised about 87.0% octaester and had an average degree of esterification of about 7.80 at 6.5 hours into the reaction.

30. I have examined Exhibit 33, and I confirm that Exhibit 33 comprises accurate copies of pages 55 and 56 of Laboratory Notebook SI 6055 on which I accurately recorded a series of experiments I conducted in March 1990, and which I signed and accurately dated upon the completion of the procedures described on each page respectively.

31. Pages 55 and 56 of Exhibit 33 describe a sucrose polyester reaction experiment which I conducted on March 26 and 27, 1990. The reaction investigated, inter alia, the effect of lower temperatures and higher pressures on the formation of DFKs in later stages of a sucrose polyester reaction. Pages 55 and 56 set forth the temperature and pressure of the reaction at thirty (30) minute intervals, beginning at 8:00 a.m. At 9:32 a.m., the reaction temperature was reduced from 135°C to 110°C and the reaction pressure was increased from 15 mm Hg to 30 mm Hg for the remainder of the reaction. Lines 13-27 of page 56 detail the percentages of the respective sucrose esters and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours into the reaction. The DFK content of the product in ppm is also set forth. As indicated on page 56, the sucrose polyester product comprised about 89.3% octaester, had an average degree of esterification of about 7.80, and comprised a DFK level of 201 ppm, all at 14.5 hours.

32. I have examined Exhibit 34, and I confirm that Exhibit 34 comprises accurate copies of pages 84 and 85 of Laboratory Notebook SI 6055 on which I accurately recorded a series of experiments I conducted in May 1990, and which I signed and accurately dated upon the completion of the procedures described on each page respectively.

33. Page 84 of Exhibit 34 describes a sucrose polyester reaction experiment which I conducted on May 16, 1990 and which was part of a matrix of experiments intended to evaluate, inter alia, the effect of the fatty acid methyl ester to sucrose molar ratio on the sucrose polyester reaction, together with the use of powdered sucrose and catalyst. The reactants comprised about 34.4 grams of powdered sucrose and about 310 grams of fatty acid methyl esters (about 146 grams initially plus about 164 grams added at 9:20 a.m.), potassium carbonate catalyst and potassium stearate soap, as described in lines 1-4 and 10 of page 84. Based upon a molecular weight of 342 for the sucrose and a molecular weight of 296 for the methyl esters, the molar ratio of methyl esters to sucrose for this experiment was about 10.4:1, which corresponds to an ester to esterifiable sites ratio of about 1.3:1. Lines 23-29 of page 84 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction and the percentage of unreacted sucrose in the reaction mixture as a function of time beginning at 1.5 hours into the reaction. As indicated on page 48, the sucrose polyester product comprised about 88.2% octaester and had an average degree of esterification of about 7.82 at 6.5 hours.

34. Page 85 of Exhibit 34 describes a sucrose polyester reaction experiment which I conducted on May 17, 1990 and which was also part of a matrix of experiments intended to evaluate, inter alia, the effect of the fatty acid methyl ester to sucrose molar ratio on the sucrose polyester reaction, together with the use of granular sucrose and catalyst. The reactants comprised about 34.4 grams of granular sucrose and about 310 grams of fatty acid methyl esters (about 146 grams initially plus about 164 grams added at 9:20 a.m.), and potassium carbonate catalyst and potassium stearate soap, as described in lines 1-4 and 10 of page 85. Based upon the

molecular weights set forth in paragraph 31, the molar ratio of ester to esterifiable sites for this experiment was also about 1.3:1. Lines 23-29 of page 85 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction and the percentage of unreacted sucrose in the reaction mixture as a function of time beginning at 1.5 hours into the reaction. As indicated on page 85, the sucrose polyester product comprised 85.7% octaester and had an average degree of esterification of 7.79 at 6.5 hours.

35. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the present application or any patent issued thereon.

Respectfully submitted,

By: Roger S Berger
Roger S. Berger

Date: 4/1/99

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